

Capabilities of the Materials Contamination Team at Marshall Space Flight Center

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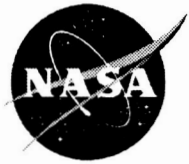
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LIST OF ACRONYMS AND SYMBOLS

AO	atomic oxygen
ASTM	American Society for Testing Materials
ATR	attenuated total reflectance
FTIR	Fourier transform infrared
IR	infrared
ISS	<i>International Space Station</i>
MPLM	multipurpose logistics modules
MP&M	Materials, Processes, and Manufacturing (Department)
MSFC	Marshall Space Flight Center
NIR	near infrared
OSEE	optically stimulated electron emission
PDA	personal digital assistant
RSRM	reusable solid rocket motor
SEM	scanning electron microscope
SOC	Surface Optics Corporation
TQCM	temperature-controlled quartz crystal microbalance
UV	ultraviolet
UVF	ultraviolet fluorescence
VASE	variable angle spectroscopic ellipsometer

TECHNICAL PUBLICATION

TEST AND ANALYSIS CAPABILITIES OF THE MATERIALS CONTAMINATION TEAM AT MARSHALL SPACE FLIGHT CENTER

1. INTRODUCTION

The Materials, Processes, and Manufacturing (MP&M) Department at Marshall Space Flight Center (MSFC) has been recognized for its contributions to NASA product lines, including space transportation, space science, and flight projects, such as the Reusable Solid Rocket Motor (RSRM), Chandra X-Ray Observatory, and the *International Space Station (ISS)*. The Environmental Effects Group, with its Materials Contamination Team and Space Environmental Effects Team, has been an integral part of MP&M success by the testing, evaluation, and qualification of materials, hardware, and processes. This Technical Publication focuses on the capabilities of the Materials Contamination Team.

The Materials Contamination Team's realm of responsibility includes establishing contamination control during all phases of hardware development, including design, manufacturing, assembly, test, transportation, launch site processing, on-orbit exposure, return, and refurbishment. The team continues its mission of reducing the risk of equipment failure due to molecular or particulate contamination. Contamination is a concern in the Space Shuttle with sensitive bondlines and reactive fluid (liquid oxygen) compatibility as well as for spacecraft with sensitive optics, such as Hubble Space Telescope and Chandra X-Ray Observatory.

The Materials Contamination Team has a variety of facilities and instrumentation capable of contaminant detection, identification, and monitoring. The team addresses material applications dealing with environments, including production facilities, clean rooms, and on-orbit exposure. The optically stimulated electron emission (OSEE) system, the ultraviolet (UV) fluorescence (UVF) surface contamination detection system, and the Surface Optics Corporation 400 (SOC 400) portable hand-held Fourier transform infrared (FTIR) spectrometer are state-of-the-art tools for in-process molecular contamination detection. The team of engineers and technicians also develop contamination calibration standards and evaluate new surface cleanliness inspection technologies. The team utilizes facilities for on-orbit simulation testing of materials for outgassing and molecular film deposition characteristics in the presence of space environmental effects, such as atomic oxygen (AO) and UV radiation exposure. Databases are maintained by the team for process materials as well as outgassing and optical compatibility test results for specific environments.

2. BACKGROUND

The Materials Contamination Team follows a general contamination control philosophy. First, determine the appropriate system requirements for materials, processes, and environments. Second, develop a contamination control program with a written plan. The contamination control program should be initiated by identifying potential contaminants, which could be molecular, particulate, or microbial, and determine the system sensitivity to those contaminants. Proper selection of materials and processes is vital to mission success. The contamination control plan should implement adequate controls over hardware processes to eliminate or control contaminants and maintain process consistency and reliability throughout the hardware lifetime. The contamination control program should include appropriate surface cleanliness inspection techniques to detect contaminants at levels that adversely affect critical systems. Witness panels or equivalent monitoring devices should be used during the actual process (not sideline) to ensure design requirements are met. All personnel who have potential contact with materials, processes, or hardware should be trained in contamination control and proper techniques for handling hardware.

Contamination can be in either particulate form, such as dust, or molecular form. Both forms can significantly degrade the performance of sensitive surfaces. Sensitive surfaces can include a wide range of applications from critical bond system substrates, such as the RSRM nozzle housings and composite liners, to highly reflective optics, with x-ray and UV optics being more sensitive than those of longer wavelengths. Contamination control includes assembly in clean rooms to minimize particulate fallout and choosing materials carefully to minimize outgassing. Spacecraft venting and engine effluent are also sources of contamination; therefore, line of sight to sensitive optics and vent design options should be considered. Molecular contamination can also impact thermal performance of radiators.^{1,2}

Particulate contamination may obscure optical systems throughout or interfere with sealing surfaces, causing higher than expected leak rates. It is much easier to control contamination throughout the fabrication and assembly of a spacecraft than to try to mitigate the effects on orbit. On January 15, 2003, astronauts Kenneth Bowersox and Donald Pettit had to perform an extravehicular activity to remove grit from a sealing ring on the Unity node on the *ISS*.³ That particular docking port is used for multipurpose logistics modules (MPLMs) that carry racks of scientific equipment and supplies to the *ISS*. Once equipment is transferred to the *ISS*, the MPLM is returned to Earth in the Space Shuttle cargo bay. The MPLM was the likely source of contamination, and removal of the particulate was necessary to allow the next MPLM to dock securely. Kapton[®] tape by DuPont[®] was used to remove the particulate.

Of particular interest to the Materials Contamination Team is the effect of molecular contamination on bonding during manufacturing of Space Shuttle solid rocket motors and external tanks. Substrate materials studied were D6ac steel, 2219-T87 and 7075-T73 aluminum, nitrile butadiene rubber insulation, and carbon and glass phenolics.⁴ Contaminant materials studied were hydrocarbons, such as Conoco HD-2 grease used as a corrosion inhibitor during RSRM case processing, and silicones used as mold release agents.

Materials must be tested for compatibility in contamination-sensitive applications, whether in processing facilities, clean rooms, or onorbit. Thermal vacuum bakeout of sensitive hardware per MSFC-SPEC-1238 and materials outgassing tests per MSFC-SPEC-1443, MSFC-SPEC-2223, and American Society for Testing Materials (ASTM) specifications of ASTM E 595 are conducted at MSFC.⁵⁻⁸ A facility for testing materials per ASTM E 1559 is also available.⁹ A facility for evaluating on-orbit contamination deposition in the presence of AO and UV is currently being characterized.

3. OPTICALLY STIMULATED ELECTRON EMISSION

The OSEE system (fig. 1) was originally developed to detect residual grease on the internal surfaces of the RSRM case after vapor degreasing.¹⁰ OSEE yields real-time data for motor case cleanliness prior to priming and adhesive application for insulation bonding. OSEE is also used to inspect the RSRM igniter chambers, systems tunnel floor plates, and nozzle housings prior to bonding. It is an excellent noncontact tool for detecting low amounts of contamination, such as grease or fingerprints on steel and aluminum substrates (fig. 2).

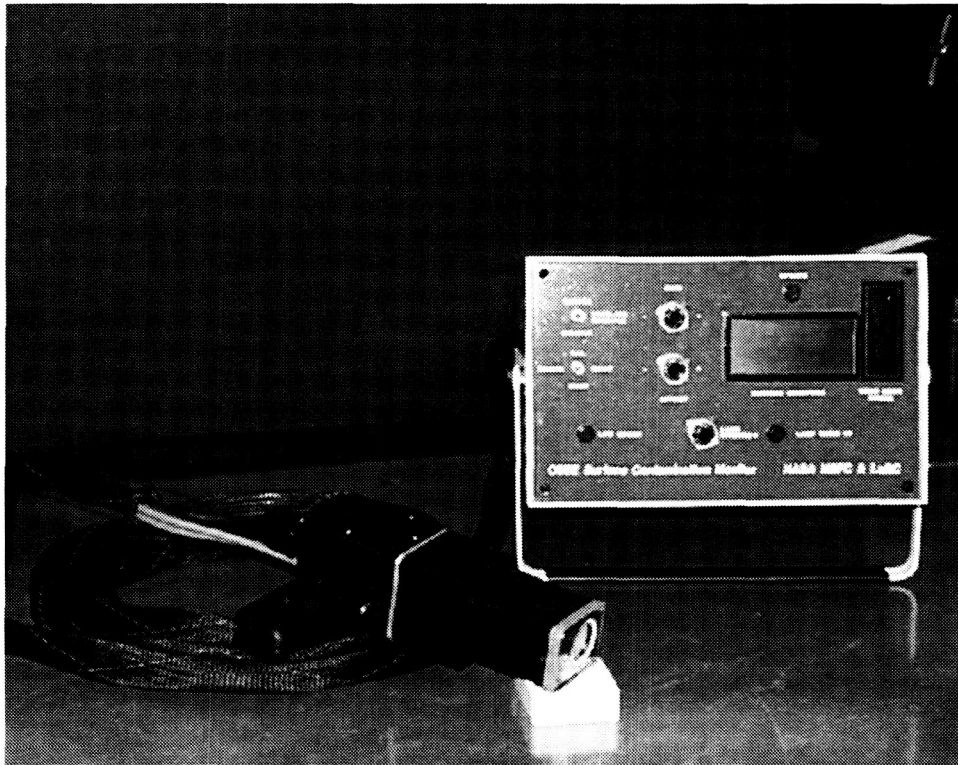


Figure 1. OSEE contamination monitor.

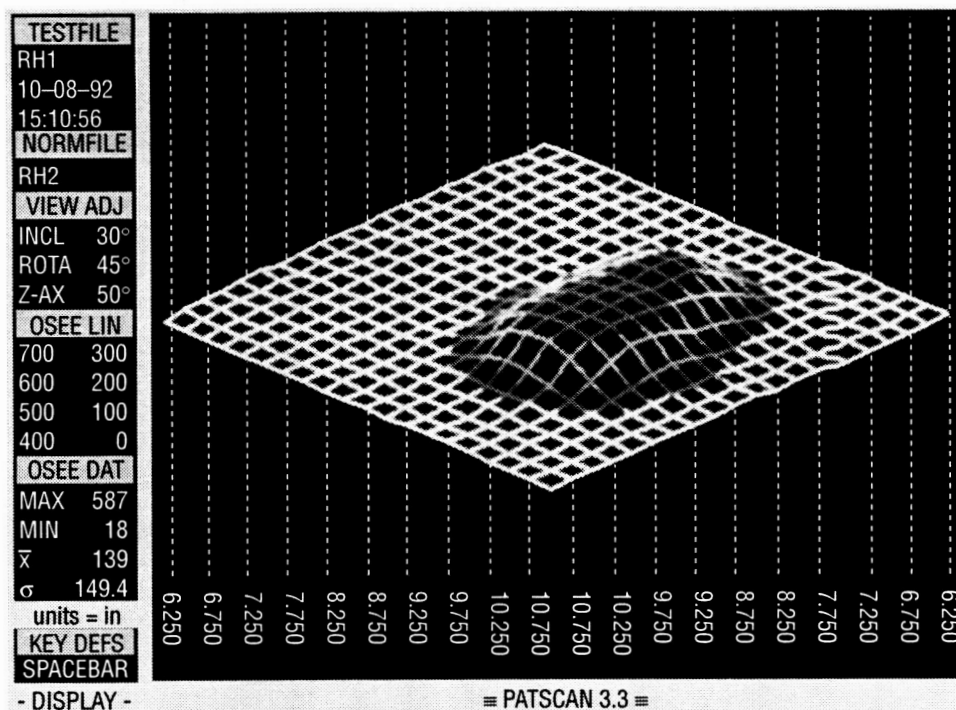


Figure 2. OSEE scan of fingerprint on aluminum panel.

OSEE illuminates the metallic test surface with UV light which induces electron emission. The sensor collector picks up the electrons. The measured electron current is in reverse proportion to the surface contamination—the heavier the contamination, the fewer photons that reach the surface and the lower the electron emission current detected.

OSEE is now in its third generation of development, with improved stability and response time. An argon purge was added in an effort to reduce effects from the ambient environment. The third-generation system is currently being characterized. Due to OSEE sensitivity, signal response can be affected by many variables. If the substrate is adequately characterized and the contaminant being detected is known, the OSEE can yield quantitative data in some cases when applied to metallic substrates.

4. ULTRAVIOLET FLUORESCENCE

The UVF system (fig. 3) uses a UV flash lamp to excite the contaminant on a surface. Contaminant fluorescence is collected by a charged-coupled device camera. Effects of ambient light are reduced by using a short flash lamp pulse of $\approx 10 \mu\text{s}$. A liquid crystal tunable filter selects, under computer control, the spectral band of fluorescence. Alternatively, an interference filter can be used to provide a higher light throughput at a particular spectral region. Intensity of the signal correlates to the amount of contamination (fig. 4).

The UVF system was developed to allow a less subjective inspection than the standard blacklight inspection commonly used. This technique will detect fluorescing contaminants at a detectable level on nonfluorescing substrates.

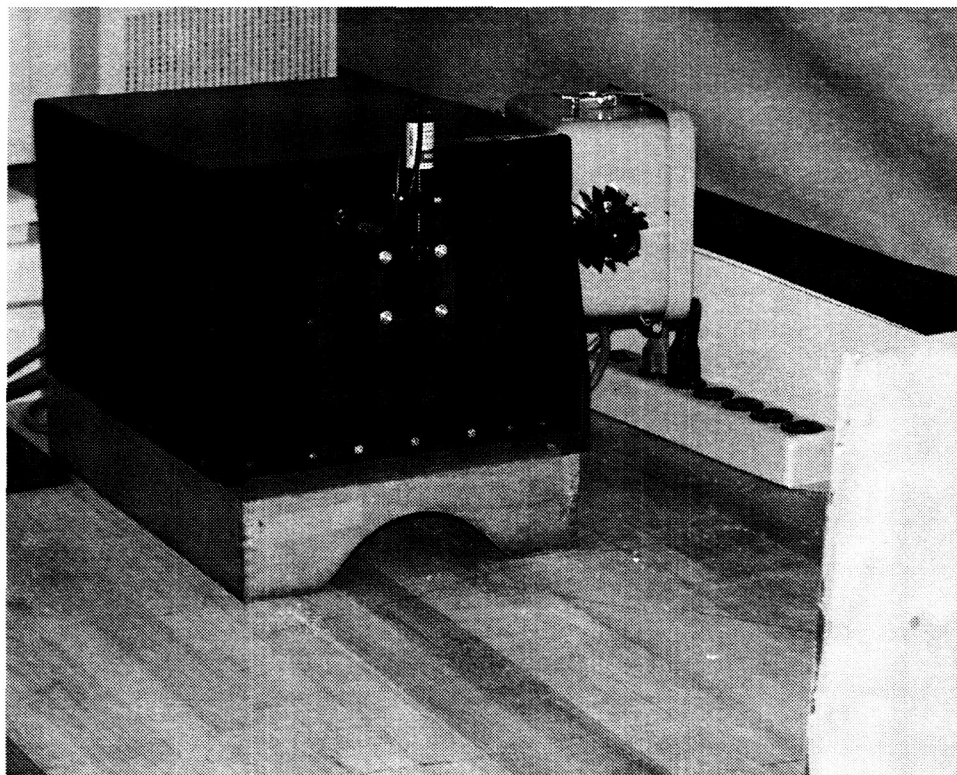


Figure 3. UVF system.

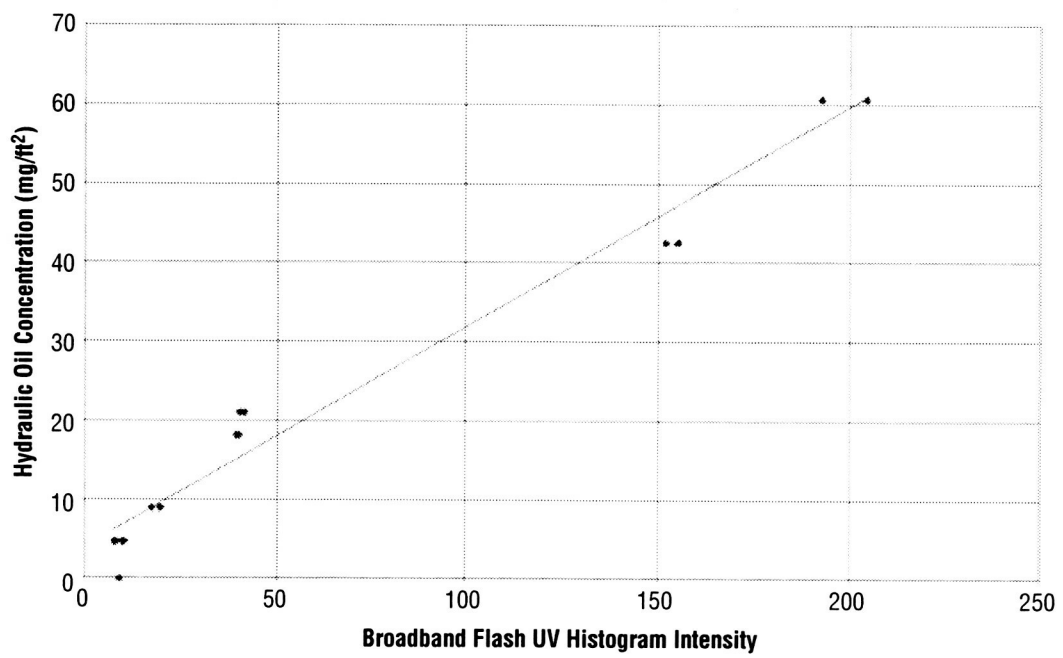


Figure 4. UVF signal versus contaminant level for hydraulic oil on steel, where $y=0.2778x + 4.0796$ and $R^2=0.9686$.

5. HIGH-INTENSITY BLACKLIGHT EVALUATION

Blacklight inspection for contamination is influenced by lamp intensity, lamp standoff distance from the substrate, and white light level. The Materials Contamination Team has high-intensity blacklights rated at 50 mW/cm^2 at an 18-in standoff. These newer blacklights are 10 times more intense than traditional blacklights. In the absence of white light, the high-intensity blacklight can be used to detect the presence of 0.5 to 1 mg/ft^2 HD-2 grease on D6ac steel, whereas the traditional blacklight is marginally effective at detecting 4 to 5 mg/ft^2 of the same contaminant.

6. NEAR-INFRARED SPECTROSCOPY

The near-infrared (NIR) spectroscopy system (fig. 5) is used for incoming material inspection and fingerprinting. Materials used in space hardware processing, such as tape, bagging material, clean room gloves, etc., are characterized to ensure consistency and detect the presence of contamination; e.g., silicone release agents. Process materials must be tightly controlled to ensure they do not adversely affect the system in process.

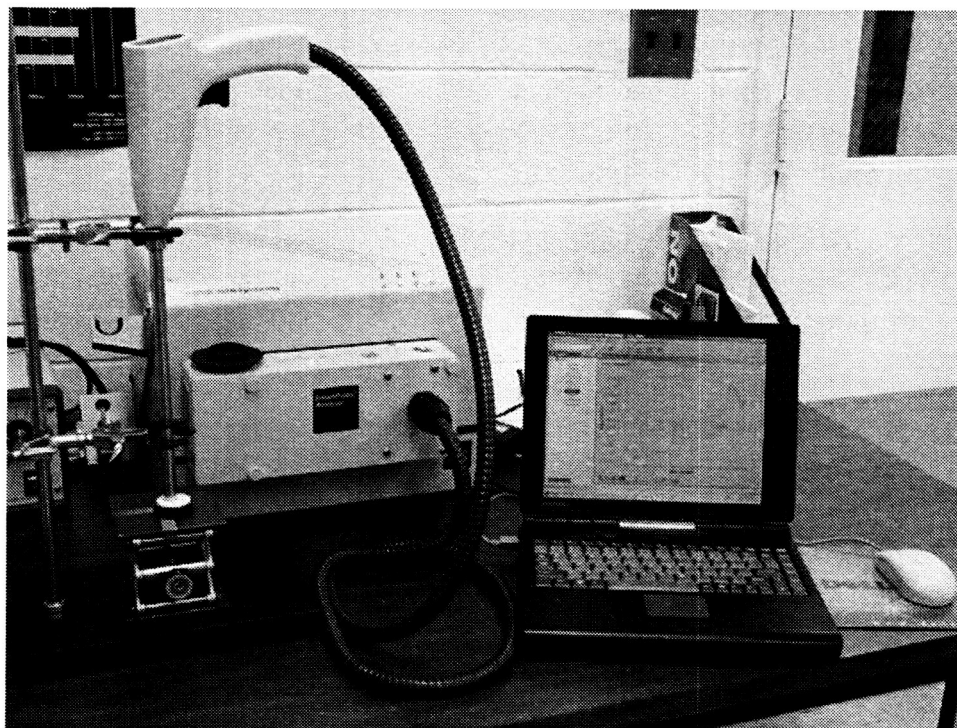


Figure 5. NIR analysis system.

7. MID-INFRARED SPECTROSCOPY

The Materials Contamination Team has several instruments for mid-infrared spectroscopy.

The Nicolet Magna 750 FTIR is a benchtop analysis system incorporating attenuated total reflectance (ATR), liquid, powder, and microscope sample accessories. Especially useful is the ATR accessory, which can easily and quickly analyze minimal (microgram) quantities of materials since it enables the infrared (IR) beam to pass through the sample multiple times. This spectrometer is used to maintain a database of IR signatures, which has been beneficial when identifying contaminants. For example, a reflectance analysis performed with the microscope IR objective successfully identified contamination collected from a solid rocket booster frustum as a hair protein. Another experiment utilized the ATR to identify a contaminant found on optical mirrors as an offgassing product from foam packaging that had been used to protect the mirrors during transport.

The SOC 400 (fig. 6) is a portable hand-held FTIR spectrophotometer with sensitivities rivaling a benchtop research FTIR microscope. Portable FTIR is also useful when surface oxidation is present. The SOC 400 measures diffuse reflectance of a surface in the 4,000- to 500-cm⁻¹ bandwidth. It can be hand held or mounted on a stationary support or a robotics arm for inspection.

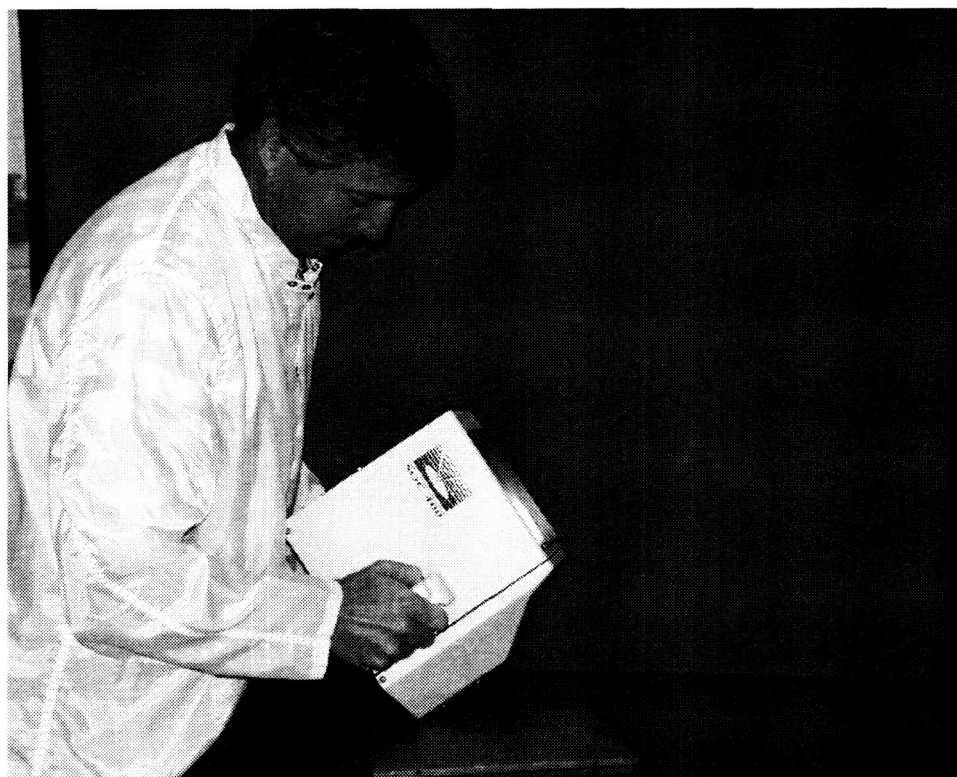


Figure 6. SOC 400 portable FTIR system.

Reflectance measurements are particularly useful when detecting the presence of a specific contaminant. For example, in one study,¹¹ eight different solvents were evaluated for removing RTV 90-006 silicone from steel. The spectrum shown in figure 7 was obtained using the SOC 400, and the 1,259-cm⁻¹ peak was used to calibrate and quantify silicone on the plate surfaces. Concentrations of silicone at 1 mg/ft² and greater showed good correlation with the spectral absorbance intensities measured.

The SOC 400 has a number of accessories that enhance its capability. A diamond attenuated total reflectance accessory can be used to analyze almost any material but is mainly used for paint and coating chemistry assessment. A diffuse reflectance head detects scattered reflection of the IR beam from rough metal or painted surfaces. Testing has shown that contamination can be detected to $\approx 0.3\text{--}0.5\text{ }\mu\text{g/cm}^2$. A specular reflectance accessory is used for smooth, reflective surfaces and can detect contamination as low as 0.2 to 0.3 $\mu\text{g/cm}^2$. Also used with smooth, reflective surfaces is the grazing angle attachment. It has a larger spot size compared to the specular reflectance head and is more sensitive, detecting contamination at $\approx 0.1\text{ }\mu\text{g/cm}^2$.

In addition to the reflectance accessories, the SOC 400 has a VSphereTM accessory, used for qualitative identification and quantitative measurement of nonvolatile residues. Surface areas are flushed with a solvent, the solvent/residue mixture is placed into the gold-plated examination cup, the solvent is allowed to evaporate, and the cup is placed directly into the VSphere head. The IR beam reflects many times inside the sample holder before it is collected. The level of contamination on a surface can be quantified by weighing the residue.

The Avatar FTIR spectrometer has analysis accessories for solids, liquids, and gases. Surface analysis can be performed using a unique fiber optic probe accessory with an ≈ 0.25 -in-diameter tip. Due to the small footprint of the probe and the flexibility of the fiber optic cable, it has been successfully used to examine otherwise inaccessible locations on rocket motor hardware.

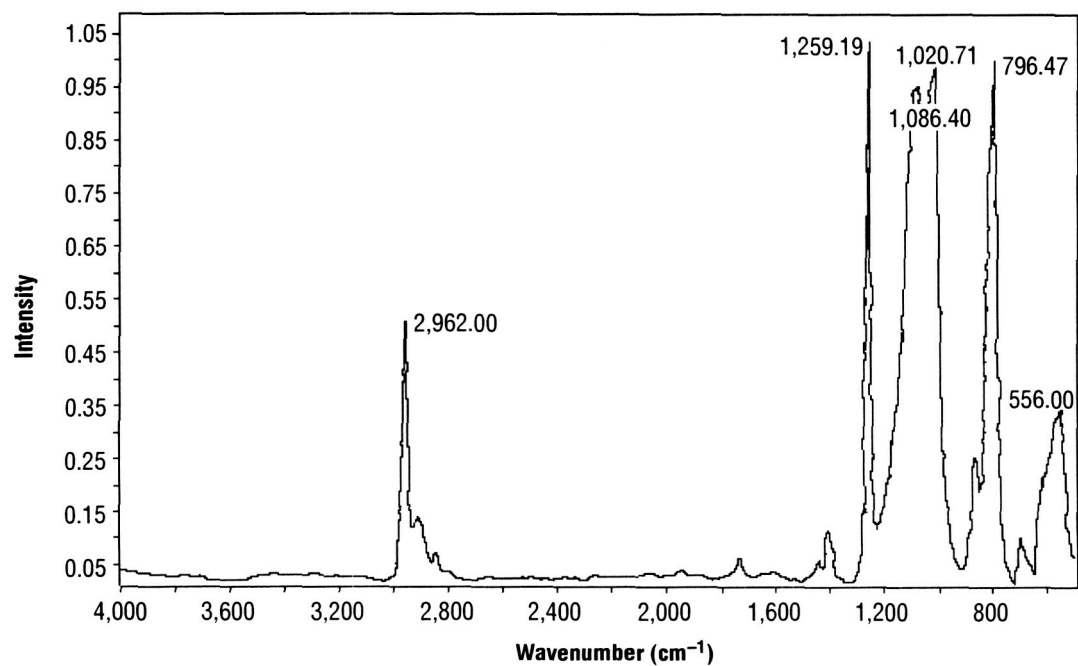


Figure 7. IR spectrum of silicone on steel plate acquired by SOC 400 spectrometer showing typical silicone peaks.

8. CONTAMINATION APPLICATION SYSTEM

The contamination application system (fig. 8) is a modified Sono-Tek spray system originally used for spraying flux on printed circuit boards. It uses an ultrasonic spray nozzle to apply low-level contaminant films. The ultrasonic nozzle has the advantage of very low flow rates and will not clog, producing consistent contaminant films from less than 0.5 up to 30 mg/ft². The Sono-Tek system was used in studying critical bond systems and their sensitivity to contamination. Sensitivity is dependent on contaminant species; e.g., silicones can be more detrimental to bonding than hydrocarbons. Bonding tests showed epoxy bondlines were sensitive to certain contaminants at levels below 5 mg/ft². RSRM vulcanized bonding for insulation was generally sensitive to ≈ 50 mg/ft² of hydrocarbon contaminant.

This system is also used to create calibration standards for contamination detection instrumentation. The development of calibration standards was required after the implementation of instruments with the capability of detecting low-level contaminants.

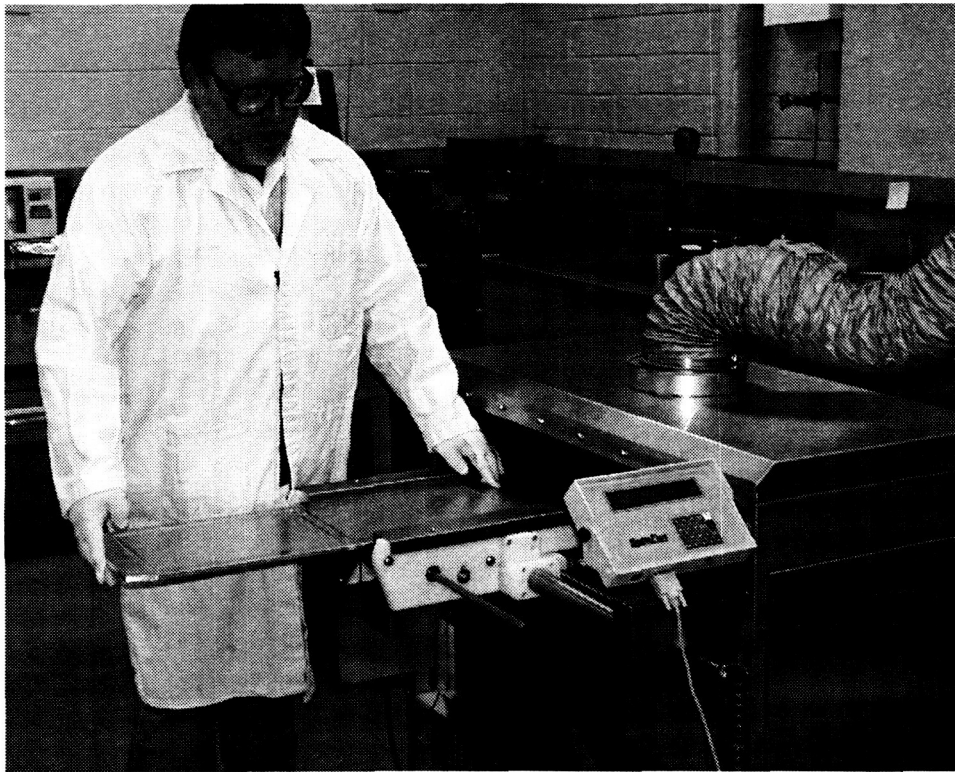


Figure 8. Contamination application (Sono-Tek) system.

9. MATERIALS OUTGASSING STUDIES

The Materials Contamination Team has facilities for characterizing the outgassing from candidate spacecraft materials and the possible impact on line-of-sight sensitive optics. At a minimum, spacecraft materials should be tested by ASTM method E 595. Total mass loss of 1 percent or more or collected volatile condensable materials of 0.10 percent or more is usually cause for rejecting the material for use on spacecraft.

MSFC-SPEC-1443 takes this a step further by placing an optical witness sample, usually a magnesium fluoride/aluminum mirror, in line of sight to the candidate material. This specification allows for testing at or slightly above the material's maximum-use temperature, rather than the standard 125 °C (257 °F) of ASTM E 595. The optical witness sample is usually held at 25 °C (77 °F), but this temperature may also be changed, depending on the expected thermal environment of the spacecraft. Reflectance in the vacuum UV wavelength range (121.6 to 200 nm) is measured. A change of ± 3 percent or more in reflectance of any wavelength is considered failure. The Materials Contamination Team maintains a database of tested materials per ASTM E 595.

MSFC-SPEC-1238 is followed for thermal vacuum bakeout of contamination-sensitive hardware. The hardware is thermally maintained just above the maximum temperature expected on orbit, while a temperature-controlled quartz crystal microbalance (TQCM) monitors the outgassing until it changes no more than 1 Hz/hr averaged over 36 hr. This translates to a deposition of 1.56×10^{-9} g/cm²/hr, assuming $\rho = 1$ g/cm³. When the TQCM is stable, an optical witness sample is cooled to 10 °C (50 °F) below the minimum expected sensitive surface temperature and exposed in the line of sight to the hardware. This optical witness sample is then examined for vacuum UV reflectance changes. Bakeout of the hardware continues until the optical witness sample passes with no more than a 3-percent change in reflectance at any vacuum UV wavelength.

MSFC-SPEC-2223 is a method for testing materials that may come in contact with sensitive optics, usually in a clean room. In the past, release films, packaging films, adhesive tape, and seal materials on shipping containers have been found to be sources of contamination. The Materials Contamination Team maintains a database of tested materials to ensure that ground handling and clean room operations will not damage sensitive optics.

The materials outgassing measurement facility at MSFC is for the measurement of molecular contaminant deposition rates per ASTM E 1559 (fig. 9). This method expands upon the ASTM E 595 outgassing measurement by measuring the deposition rate of molecular species, providing deposition rate data that can be used by computer models to predict the buildup of volatile, condensable materials of spacecraft external surfaces. Four cryogenic quartz crystal microbalances are used to independently measure the rate of deposition on surfaces from 80 °C (176 °F) to -100 °C (-148 °F), while the material sample is heated in an effusion cell.

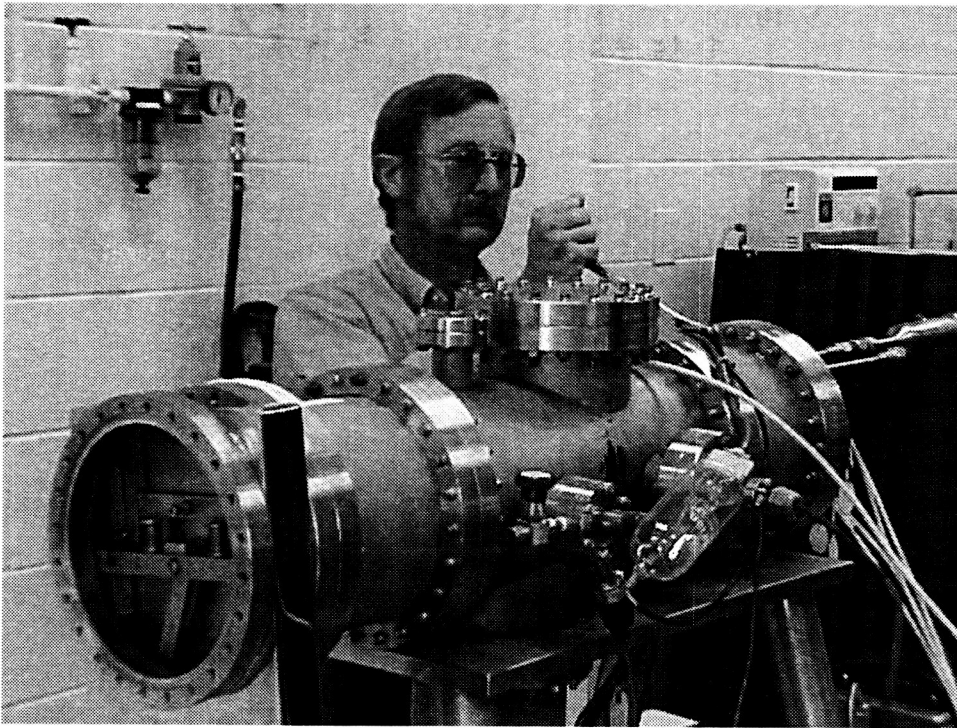


Figure 9. Materials outgassing test facility per ASTM E 1559.

10. PHOTODEPOSITION CHAMBER

The deposition of volatile, condensable materials on a surface can be altered by UV radiation interaction with the molecular species^{12,13} that comprise the contaminant(s). The rate of deposition can be enhanced by photopolymerization of the molecules and the fixing of the material to the substrate through the formation of chemical bonds. Atomic oxygen can convert some materials, such as silicones, to a silicate layer that is extremely difficult to remove or destructively interact with the molecular species, such as a hydrocarbon, to remove the contaminant.¹⁴

The MSFC photodeposition chamber (fig. 10) has been constructed to study the individual and combined effects of UV and AO on volatile, condensable materials. The chamber has both a TQCM and an optical witness mirror for monitoring deposition. Other samples, such as white thermal control coatings or thin polymer films, can be placed in the chamber for exposure to the outgassed contaminant. Samples of materials used in spacecraft construction are heated in an effusion cell to produce the contamination. A deuterium UV lamp (wavelength range of 115 to 400 nm) is positioned in the vacuum chamber so that it can illuminate the deposition surface during the molecular deposition process. In addition, a source of AO is also aligned with the deposition surface of the TQCM. The system can be operated to measure the individual parameters or any combination of the three processes. By varying the parameters, the effects of UV and AO on the deposition of molecular species can be quantified, providing rate data that are used as input for spacecraft contamination models and more representative of the on-orbit environment.

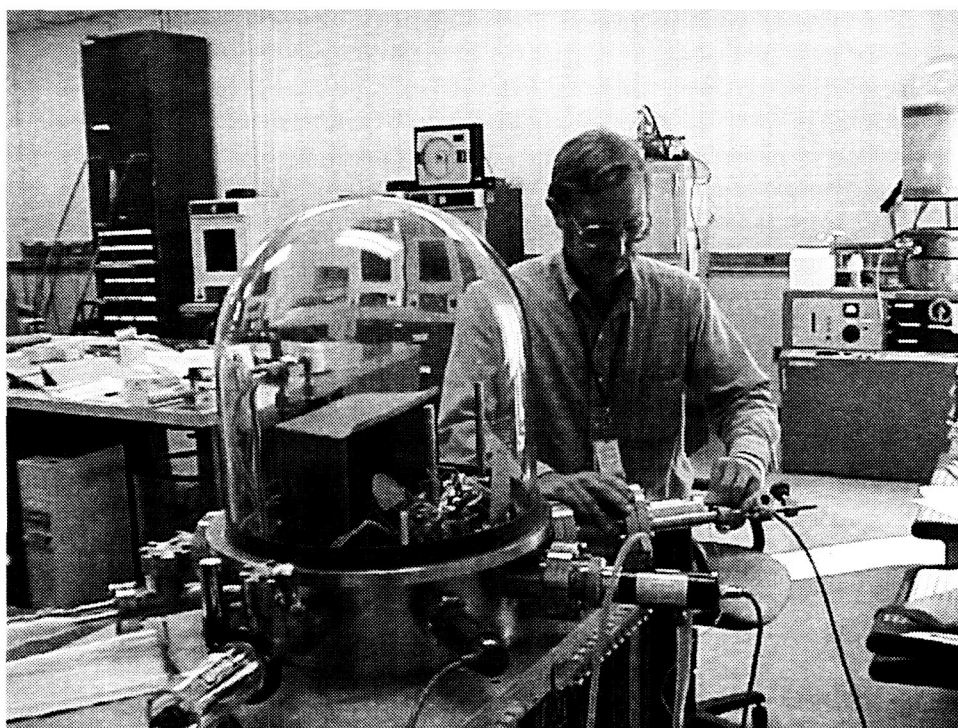


Figure 10. Photodeposition chamber.

11. ELLIPSOMETRY

An ellipsometer measures the change in the polarization state of light reflected from a surface. The change in polarization can be quantified through the index of refraction, n , and the extinction coefficient, k , of the surface. The thickness and optical constants of any contamination on the surface of the material also change the light polarization.

The variable angle spectroscopic ellipsometer (VASE) (fig. 11) is a useful tool for analyzing mirrors or other smooth, reflective surfaces for contamination. Greater sensitivity is obtained by changing the angle of incidence and varying the light wavelength from near UV to near IR during sample scans. The VASE scans from 240 to 1,100 nm at angles of incidence varying from 12° to 90° . It can analyze thin films ranging in thickness from 10 to 5,000 Å on optical surfaces. In addition to molecular films, thin layers of metals can be sampled and characterized.

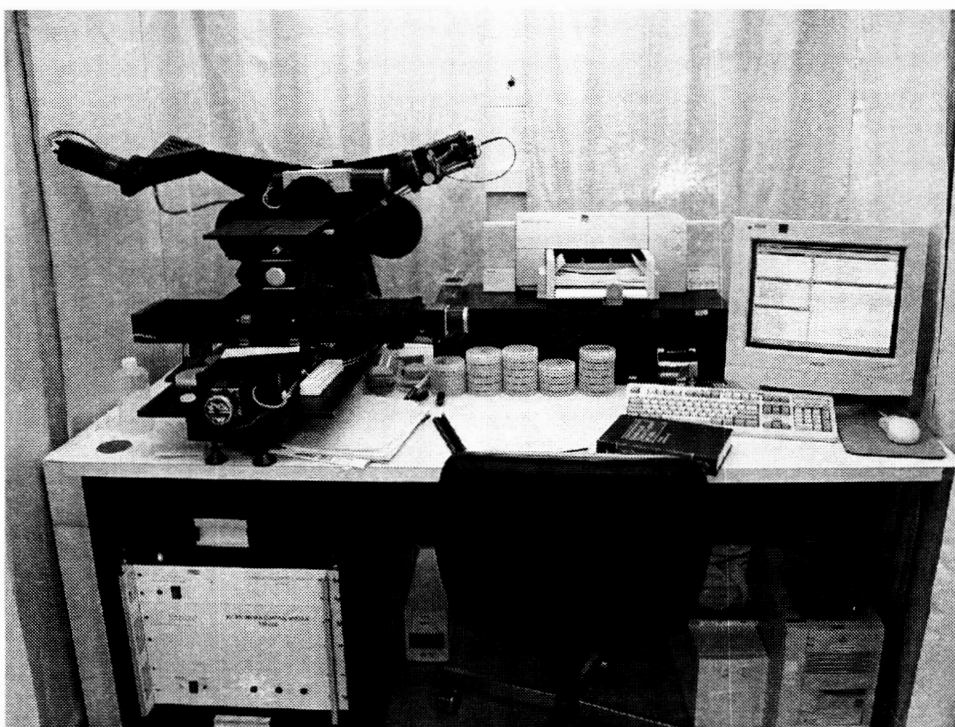


Figure 11. Variable angle spectroscopic ellipsometer.

12. PARTICULATE ANALYSIS SYSTEM

The particulate analysis system consists of an optical microscope with a motor-controlled stage and video camera, the stage controller, and a computer with image analysis software (fig. 12). A sample surface is scanned for particulate contamination, with particles sorted by size (down to 5 μm) and the total number of particles counted. From these data, the amount of obscuration is calculated. Using the imaging system and software, other types of dimensional operations besides particulate analysis may be performed.

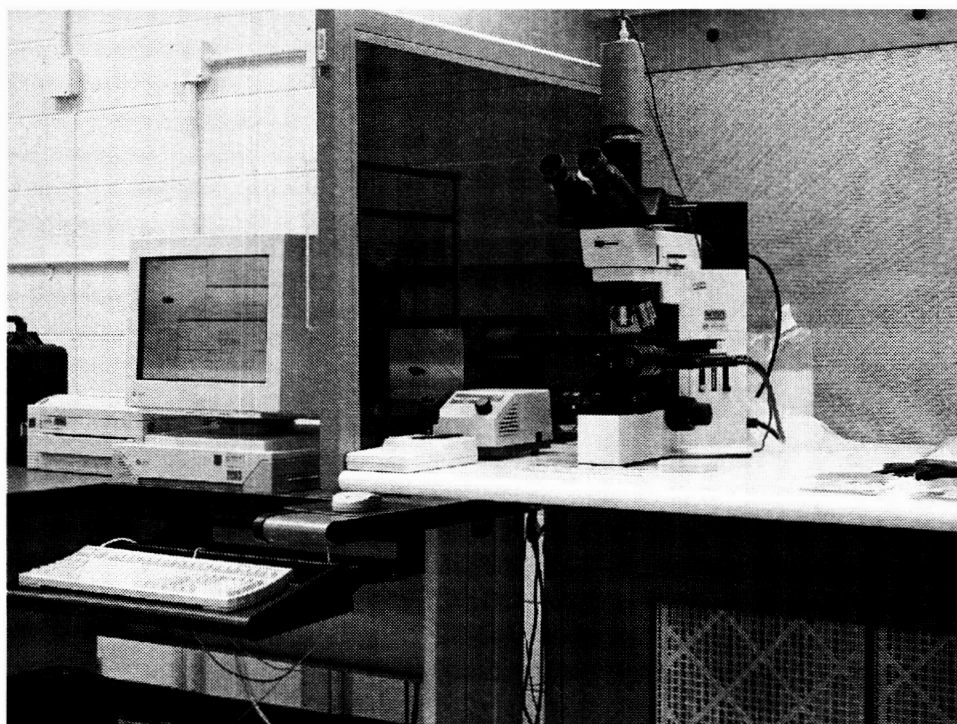


Figure 12. Image analyzer.

13. HAND-HELD X-RAY FLUORESCENCE ANALYZER

The TRACeR III-V is a portable, wide-range elemental analyzer intended mainly for metal alloy analysis applications (fig. 13). The unit is based on energy dispersive x-ray fluorescence technology and uses an x-ray tube as its excitation source. The TRACeR III-V provides a method for chemical analysis or sample identification (sorting) directly from samples of various forms. The instrument is comprised of a fully field portable analyzer with an integrated personal digital assistant (PDA) computer and vacuum system that allows elemental detection down to aluminum. Within the TRACeR III-V analysis program on the PDA, the user may select analytical modes, view spectra, and save data. The instrument is factory calibrated for aluminum, titanium, low alloy steels, stainless steels, tool steels, nickel alloys, cobalt alloys, and copper alloys.

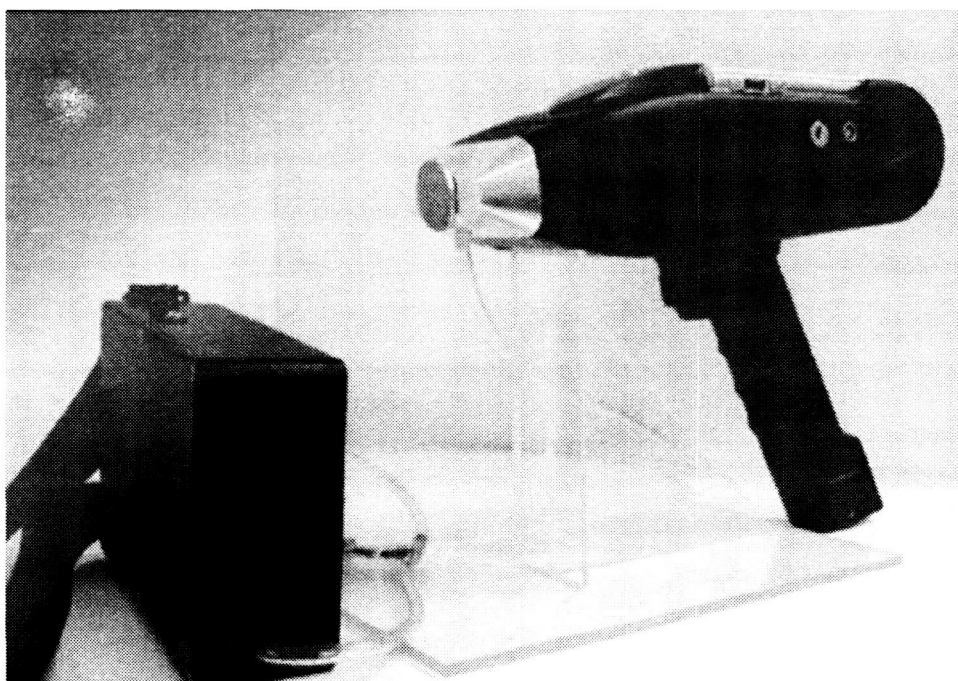


Figure 13. TRACeR III-V analyzer.

14. OTHER ANALYTICAL CAPABILITIES

The Materials Contamination Team has many capabilities for materials characterization. Optical properties, such as spectral transmission, solar absorptance, infrared emittance, and bidirectional reflectance distribution function, can be measured. Spectral transmission is measured using a Perkin-Elmer Lambda 19 spectrophotometer for 185–2,500 nm. Reflectance or transmission measurements in the vacuum UV wavelengths of 120–300 nm can be made with a McPherson 0.5-m scanning monochromator with a deuterium lamp. Solar absorptance is measured for the wavelength range of 250 to 2,800 nm using an AZ Technology laboratory portable spectrophotometer. Infrared reflectance measurements are made using an AZ Technology TEMP 2000 Infrared Reflectometer or an AZ Technology laboratory portable infrared reflectometer for spectral measurements between 2.5 and 20 μ wavelengths. The bidirectional reflectance distribution function is measured using a TMA Quickscan with detectors for measuring reflectance at 632.8 nm at up to 10 different angles. Microgram mass changes of samples can be measured.

The Materials Contamination Team also has a scanning electron microscope (SEM), which can be used to conduct particle size measurements and to evaluate defects. The SEM has also been used to evaluate microballoon integrity in adhesives and postflight changes in surface morphology of atomic oxygen-exposed samples.

15. DISCUSSION AND CONCLUSIONS

The world-class facilities of the Environmental Effects Group at MSFC provide valuable information to designers, engineers, and scientists on materials and processes related to contamination control. Contamination control is needed for mission success. The Materials Contamination Team works with the aerospace industry to improve methods for detection and elimination of contaminants from spacecraft processing. The test facilities presented here have also been invaluable in postflight analysis of the Long Duration Exposure Facility, the Passive Optical Sample Assembly, the Space Portable Spectroreflectometer, and the Optical Properties Monitor.

The Materials Contamination Team also advises on clean room operations by compliance with MSFC-STD-246B. This standard lists guidelines for selecting environmental facilities and for achieving the operating conditions necessary to achieve contamination control of critical aerospace hardware during fabrication, assembly, integration, transportation, overhaul, or repair; it also lists quality control measures to ensure compliance.

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13. ABSTRACT (Maximum 200 words) <p>The Materials Contamination Team of the Environmental Effects Group, Materials, Processes, and Manufacturing Department, has been recognized for its contribution to space flight, including space transportation, space science, and flight projects, such as the reusable solid rocket motor, Chandra X-Ray Observatory, and the <i>International Space Station</i>. The Materials Contamination Team's realm of responsibility encompasses all phases of hardware development including design, manufacturing, assembly, test, transportation, launch-site processing, on-orbit exposure, return, and refurbishment if required. Contamination is a concern in the Space Shuttle with sensitive bondlines and reactive fluid (liquid oxygen) compatibility as well as for sensitive optics, particularly spacecraft such as Hubble Space Telescope and Chandra X-Ray Observatory.</p> <p>The Materials Contamination Team has a variety of facilities and instrumentation capable of contaminant detection, identification, and monitoring. The team addresses material applications dealing with environments, including production facilities, clean rooms, and on-orbit exposure. The team of engineers and technicians also develops and evaluates new surface cleanliness inspection technologies. Databases are maintained by the team for process materials as well as outgassing and optical compatibility test results for specific environments.</p>				
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